IS 5062-4 (2004): Methods of test for brown coals and lignites, Part IV: Determination of the yield of benzene soluble extract [PCD 7: Solid Mineral Fuels]

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IS 5062 ( Part 4 ) : 2004
ISO 975 : 2000

भारतीय मानक

# भूरा कोयला और लिग्नाइट की परीक्षण पद्धतियाँ

## भाग 4 बेंज़ीन-विलेय निष्कर्ष की यील्ड ज्ञात करना — अर्द्धस्वचालक पद्धति

( पहला पुनरीक्षण )

*Indian Standard*

# METHODS OF TEST FOR BROWN COALS AND LIGNITES

## PART 4 DETERMINATION OF YIELD OF BENZENE-SOLUBLE EXTRACT — SEMI-AUTOMATIC METHOD

*( First Revision )*

ICS 75.160.10



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*November* 2004

**Price Group 2**

NATIONAL FOREWORD

This Indian Standard ( Part 4 ) ( First Revision ) which is identical with ISO 975 : 2000 'Brown coals and lignites — Determination of yield of benzene-soluble extract — Semi-automatic method' issued by the International Organization for Standardization ( ISO ) was adopted by the Bureau of Indian Standards on the recommendations of the Solid Mineral Fuels Sectional Committee and approval of the Petroleum, Coals and Related Products Division Council.

This Indian Standard was first published in 1969 which was essentially based on the 'Draft Recommendation No. 1126' issued by the International Organization for Standardization ( ISO ). Since this draft recommendation has subsequently been published as ISO 975 : 1975 and revised in 1985 and 2000, the Committee has decided to revise this standard to completely align it with ISO 975 : 2000 under dual number standard. Consequently the designation and title of the standard has been modified as follows:

IS 5062 ( Part 4) /ISO 975 : 2000 Methods of test for brown coals and lignites: Part 4 Determination of yield of benzene-soluble extract — Semi-automatic method

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma ( , ) has been used as a decimal marker while in Indian Standards, the current practice is to use a point ( . ) as the decimal marker.

In this adopted standard reference appears to one International Standard for which no Indian Standard exists.

The Technical Committee responsible for the preparation of this standard will review the provisions of ISO 5068 and will decide whether it is acceptable for use in conjunction with this standard.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'.

*Indian Standard*

# METHODS OF TEST FOR BROWN COALS AND LIGNITES

## PART 4 DETERMINATION OF YIELD OF BENZENE-SOLUBLE EXTRACT — SEMI-AUTOMATIC METHOD

*( First Revision )*

## 1 Scope

This International Standard specifies a semi-automatic method for determination of the yield of benzene-soluble extract in brown coals and lignites.

## 2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For a dated reference, subsequent amendments to, or revisions of, the publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For an undated reference, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

## 3 Principle

A test portion of the brown coal or lignite is extracted with benzene in a semi-automatic extraction instrument. The solvent is then removed by evaporation and the soluble residue dried to constant mass. The percentage of benzene-soluble extract is calculated from the mass of residue after drying and is reported on the dry basis.

## 4 Reagent

**Benzene**, of analytical reagent grade, $\rho_{20}$ = 0,876 g/ml, distillation range 80 °C to 81 °C. At least 95 % shall distil within this range.

**WARNING — Benzene is flammable and toxic by inhalation, ingestion or skin absorption.**

**The test must be carried out in a hood and the benzene must be recovered as completely as possible.**

## 5 Apparatus

**5.1 Semi-automatic extraction instrument,** containing mainly two units: the continuous extraction-evaporation device and the controller. The continuous extraction-evaporation device consists of a 100 ml conical flask, an extraction chamber and a condenser. The extraction chamber is 180 mm long and 30 mm in internal diameter and is provided with a water jacket through which the bath water is circulated in order to maintain the extraction temperature around the extraction chamber.

**5.2 Extraction thimble,** 25 mm × 80 mm. Cellulose or other thimbles are purchased or made as follows.

Cut filter paper into pieces of 75 mm × 75 mm and 25 mm × 25 mm. Moisten one large piece of the filter paper with distilled water and roll it snugly onto the external wall of a test tube of 25 mm diameter with a small hole pierced at the bottom. A small piece of filter paper is next moistened and rolled onto the bottom. Three large pieces and two small pieces are then rolled alternately onto the test tube. Remove the formed moist thimble by blowing at the mouth of the test tube and dry it in air or in an oven at 100 °C.

5.3 **Air oven,** capable of maintaining a temperature between 105 °C and 110 °C, or **vacuum oven**, electrically heated, in which a temperature of 80 °C ± 2 °C and a pressure of about 5 kPa can be maintained.

5.4 **Analytical balance,** accurate to 0,1 mg.

## 6 Preliminary adjustment of instrument

### 6.1 Adjustment of the extraction temperature

Place an extraction thimble with about 2 g of coal sample in the extraction chamber. Add 60 ml to 70 ml of benzene to the conical flask. Connect the flask to the extraction chamber. Switch on the electrical power and push down the programme button. The extraction-evaporation device will then be automatically lowered until the flask is immersed in the water bath and the water bath starts heating. As the first drop of condensed benzene drips from the condenser, adjust the temperature of water bath so that the dripping rate of benzene is about 4 ml/min to 5 ml/min and the sample is completely immersed in benzene in the thimble. Record the temperature and fix the position of the temperature controller. This temperature should be readjusted for changed ambient temperature.

### 6.2 Selection of periods of extraction, rinsing and evaporation

In general, the suitable periods of the above three steps are respectively 180 min, 10 min and 50 min. They can be readily adjusted with the corresponding timers. In the case of a high content of benzene-soluble constituents in the sample or excessively low barometric pressure, etc., readjustment of the extraction period may be necessitated in order to ensure the correct end-point of extraction, which is to be judged by the colourlessness of the last drops of extract solution.

### 6.3 Procedure

Weigh, to the nearest 0,2 mg, about 2 g of general analysis test sample, transfer to the extraction thimble (5.2) and cover with a pad of absorbent cotton, which is fitted snugly on the wall of the thimble.

Place the extraction thimble with sample in the extraction chamber (see 5.1).

Add 60 ml to 70 ml of benzene to a previously dried and accurately weighed flask.

Assemble the instrument.

Switch on the electrical power. Push down the programme button. The instrument will automatically perform the experiment in accordance with the following sequence:

The extraction-evaporation device is lowered until the flask is immersed in the water bath as in the preliminary adjustment and the condenser is in an inclined position permitting refluxing. The heating of the water bath is started simultaneously.

When the extraction temperature previously set is reached, the pump begins to circulate the hot water between the bath and the jacket of the extraction chamber. The benzene vapour from the flask passes through the extraction chamber and reaches the condenser, where it is condensed and drips onto the thimble. The extraction stage is thus in progress.

After 180 min, or an otherwise set time, the pump stops and extraction is finished. Hot water flows back to the bath. The temperature of the extraction chamber drops to a temperature at which the benzene vapour can only reach the extraction chamber and condenses there. Thus a rinsing action is achieved, by means of which the benzene-

soluble extract adhered to the wall is washed down into the flask by the condensed benzene. This is the rinsing stage.

After 10 min, rinsing is finished. The condenser is automatically changed to an inverted position permitting distillation. The pump works again so as to resume hot-water circulation. The benzene vapour condenses in the condenser and flows to the receiver. The stage of evaporation starts.

After 50 min of evaporation is finished, the extraction-evaporation system is elevated to the original position, permitting the flask to be detached from the extraction chamber. The programme is thus terminated.

Detach the flask with soluble residue. Dry it in the air oven (5.3) maintained at 105 °C to 110 °C or in the vacuum oven maintained at 80 °C ± 2 °C and about 50 kPa to constant mass.

NOTE Constancy in mass is considered to have been achieved when the difference between successive dryings does not exceed 0,001 g.

Carry out a moisture determination on a separate test portion by the method specified in ISO 5068.

## 7 Expression of results

The yield of benzene-soluble extract, $w_{E,ad}$, in the general analysis test sample, expressed as a percentage by mass, is given by the formula

$$w_{E,ad} = \frac{m_1 \times 100}{m_2}$$

where

$m_1$ is the mass, in grams, of benzene-soluble extract;

$m_2$ is the mass, in grams, of the test portion.

The yield, expressed on the dry basis, is given by the formula

$$w_{E,d} = \frac{100}{100 - w_M} \times w_{E,ad}$$

Where $w_M$ is the mass fraction of moisture, in percent, of the general analysis test sample.

The result (the mean of duplicate determinations, see 8.1) shall be reported on the dry basis to the nearest 0,1 %.

## 8 Precision of the method

### 8.1 Repeatability limit

The results of duplicate determinations, carried out at different times within a short interval, in the same laboratory, by the same operator, with the same apparatus, on two representative test portions taken from the same analysis sample, should not differ by more than the values shown in Table 1.

### 8.2 Reproducibility critical difference

The means of the results of duplicate determinations, carried out in each of two different laboratories, on representative test portions taken from the same sample after the last stage of sample preparation, should not differ by more than the values shown in Table 1.

**Table 1**

| **Yield of benzene-soluble extract** % (*m*/*m*) | **Repeatability limit** (air-dried basis) | **Reproducibility critical difference** (dry basis) |
|---|---|---|
| less than 5 | 0,3 % absolute | 0,5 % absolute |
| 5 to 10 inclusive | 0,5 % absolute | 0,7 % absolute |
| more than 10 | 5 % of the mean result | 7 % of the mean result |

## 9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) identification of the product tested;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in ISO 5068, or regarded as optional.

## Bureau of Indian Standards

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## Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 7 ( 2048 ).

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
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